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 The harmful living thing prevention-of-the-breeding-and-extermination agent containing oxime ether and this
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 (22) Application
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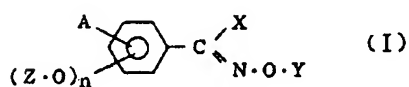
Specification

1. Name of Invention

The harmful living thing prevention-of-the-breeding-and-extermination agent containing oxime ether and this

2. Claim

(1)
 oxime ether shown by the general formula (I).



Inside of [type,
 A is hydrogen,
 hydroxy,
 methoxy,
 ethoxy,
 Halogen,
 X is hydrogen,
 methyl,
 Ethyl,
 phenyl,
 p- fluorophenyl is expressed,
 When Y is 2- fluoro ethyl,
 Z is alkyl of C1-C4,

haloalkyl of C1-C4,
 alkenyl of C2-C4,
 haloalkenyl of C2-C4,
 phenyl,
 halo substitution phenyl,
 N and N- dimethylsulfamoyl,
 It is chosen out of N and N- dimethylcarbamoyl,
 When Z is 2- fluoro ethyl,
 Y is hydrogen,
 alkyl of C1-C4,
 haloalkyl of C1-C4 (except for 2- fluoro ethyl),
 alkenyl of C2-C4,
 haloalkenyl of C2-C4,
 group chosen from alkoxycarbonylmethyl is expressed,
 n expresses 1 or 2.]

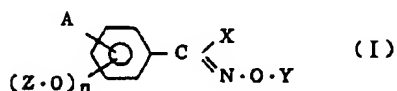
(2)

The harmful living thing prevention-of-the-breeding-and-extermination agent which consists of the oxime ether shown by the general formula (I) of claim 1 publication, an inactive dilution agent, or a carrier.

3. Detailed Explanation of Invention

(Field of the Invention)

This invention relates to the oxime ether shown by the general formula (I), and the harmful living thing prevention-of-the-breeding-and-extermination agent containing the compound concerned in more detail about the harmful living thing prevention-of-the-breeding-and-extermination agent containing new oxime ether and new it.



Inside of [type,
 A is hydrogen.
 hydroxy,
 methoxy,
 ethoxy,
 Halogen,
 X is hydrogen,
 methyl,
 Ethyl,
 phenyl,
 p- fluorophenyl is expressed,
 When Y is 2- fluoro ethyl,
 Z is alkyl of C1-C4,

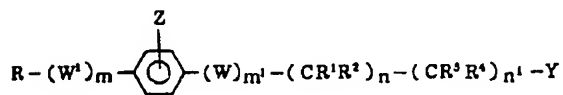
haloalkyl of C1-C4,
 alkenyl of C2-C4,
 haloalkenyl of C2-C4,
 phenyl,
 halo substitution phenyl,
 N and N- dimethylsulfamoyl,
 It is chosen out of N and N- dimethylcarbamoyl,
 When Z is 2- fluoro ethyl,
 Y is hydrogen,
 alkyl of C1-C4,
 haloalkyl of C1-C4 (except for 2- fluoro ethyl),
 alkenyl of C2-C4,
 haloalkenyl of C2-C4,
 group chosen from alkoxycarbonylmethyl is expressed,
 n expresses 1 or 2.]

(The conventional technology)

Although many medicines are used to the damage by blight and harmful insects of crops on the occasion of cultivation of plantation art crops, the prevention-of-the-breeding-and-extermination effect is insufficient, or the noxious insect and fungus of medicine resistance appear, use of the medicine is restricted, or the toxicity over men-and-beasts fishes is strong, and there is not little what is hard to be called harmful living thing prevention-of-the-breeding-and-extermination agent which should not necessarily be satisfied.

a former and oxime ether system harmful living thing
 prevention-of-the-breeding-and-extermination agent -- various proposals -- it is -- for example
 Japanese Patent Publication No. 47-No. 17994,
 Japanese Patent Publication No. 51-No. 26483,
 Provisional Publication No. 54-No. 141740,
 Provisional Publication No. 59-No. 193862,
 United States Patent No. 4079149
 And there is Provisional Publication No. 61-No. (United States Patent No. 4647698) 72733 etc.

Although Provisional Publication No. 61-No. 72733 has the extensive proposal shown by the following formula among these, the noxious insect control effect is not shown concretely.



When an activity comparison examination is carried out about the illustrated typical compound (compound No.2) to various noxious insects (lotus mandarin orange HADANI, HIMETOBIIUNKA, MONYOTOU and KONAGA) for being concretely desirable (this example of specification examination 5 reference), the region which should still be enough satisfied from a viewpoint of activity is not arrived at.

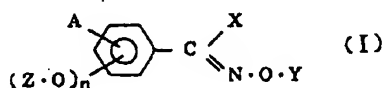
(Object of the Invention)

These inventors conquer the fault which the conventional medicine has in the basis of such a situation, and are to find out the medicine which can be safely used to the men and beasts which show the effect stabilized also on outdoor conditions.

(Means for solving a subject)

As a result of inquiring by compounding many compounds with careful attention to the above-mentioned fault, these inventors find out that the prevention-of-the-breeding-and-extermination effect in which the compound group shown by the above-mentioned general formula (I) was excellent to various harmful living things is shown, complete this invention, and came to offer the harmful living thing prevention-of-the-breeding-and-extermination agent new here.

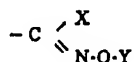
The compound group which becomes this invention is oxime ether shown by the general formula (I),



Inside of [type,
 A is hydrogen,
 hydroxy,
 methoxy,
 ethoxy,
 Halogen,
 It is hydrogen preferably,
 Chlorine is expressed,
 X is hydrogen,
 methyl,
 Ethyl,
 phenyl,
 p- fluorophenyl,
 It is hydrogen preferably,
 methyl,
 Ethyl is expressed,
 When Y is 2- fluoro ethyl,
 Z is alkyl of C1-C4,
 haloalkyl of C1-C4,
 alkenyl of C2-C4,
 haloalkenyl of C2-C4,
 phenyl,
 halo substitution phenyl,
 N and N- dimethylsulfamoyl,

N and N- dimethylcarbamoyl,
 It is an iso propyl preferably,
 2- fluoro ethyl,
 allyl,
 2- butenyl,
 3- chloro -2- propenyl,
 It is chosen out of N and N- dimethylsulfamoyl,
 When Z is 2- fluoro ethyl,
 Y is hydrogen,
 alkyl of C1-C4,
 haloalkyl of C1-C4 (except for 2- fluoro ethyl),
 alkenyl of C2-C4,
 haloalkenyl of C2-C4,
 alkoxy carbonylmethyl,
 It is an iso propyl preferably,
 allyl,
 2- butenyl,
 group chosen from 3- chloro -2- propenyl is expressed,
 n is 1 or 2,
 Although 1 is expressed preferably,
 As an especially desirable compound group,
 A is hydrogen,
 X is hydrogen, methyl, and ethyl,
 Y and Z are 2- fluoro ethyl simultaneously,
 It is the case where n is 1.]

These have the feature in Y, Z or that it is 2- fluoro ethyl simultaneously, and group shown in -OZ group and the following being directly linked with a benzene ring simultaneously,



Although it is new reference non-indicated substance, the effect attains to the harmful living thing covering the large range.

especially -- a lotus -- the lepidopteran noxious insect represented by MONYOTOU, KONAGA, etc.,
 Half-wing noxious insects, such as HIMETOBIUNKA and a MOMOAKA plant louse,
 It has the prevention-of-the-breeding-and-extermination effect excellent in HADANI represented by NAMIHADANI, mandarin orange HADANI, can ZAWAHADANI, etc.

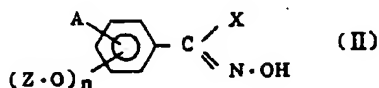
By the annual use of a medicine same especially in recent years, an effect finds out that it is highly useful practically also for the harmful living thing of the so-called organophosphorus pesticide with which the prevention-of-the-breeding-and-extermination effect fell, and synthetic pyrethroid agent resistance, and it came to complete this invention.

Next, the manufacturing process is explained in detail.

Although this invention compound shown by the general formula (I) can be manufactured by the following methods, it is not limited to this.

(1)

This invention compound shown by the general formula (I) can be obtained by making it react with compound Y' under existence of the compound shown by the general formula (II) and a base.



(AX and Z are the same as the above among a formula.)

Compound Y

(Y' is haloalkyl of C1-C4,
dihaloalkyl of C1-C4,
haloalkenyl of C2-C4,
dihaloalkenyl of C2-C4,
alkoxycarbonylhalomethyl is expressed.

Specifically as compound Y', it is,
Iodation methyl,
Ethyl bromide
2- bromo propane,
sec- butylchloride,
1- bromo -2- fluoro ethane,
1- bromo -3- chloro -2- methyl propane,
allyl bromide,
1- chloro -2- butene,
4- bromo -1- butene,
1, 3- dichloro propene,
methoxycarbonylchloromethyl etc. can be raised.

The quantity of the trial agent with which a reaction is presented is 1-1.5Eq about 1-1.5Eq of bases, and Y', and should just make these react with the reaction temperature of 10-80DEGC among a solvent to 1Eq of compounds of a general formula (II) for 1-24 hours.

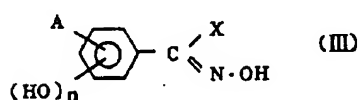
As a base,
Sodium methoxide,
Sodium ethoxide,
Sodium carbonate,
Potassium carbonate,
Sodium hydroxide,
Water oxidization potassium etc. can be raised.

moreover -- as a solvent -- for example
 Methanol,
 Ethanol,
 dioxane,
 N and N- dimethylform amide,
 A tetra hydro furan etc. can be raised.

After a reaction end can perform the usual post-operation, can remove water-soluble discard etc.,
 and can refine it by chromatography, distillation, etc. if needed.

(2)

When Y and Z are 2- fluoro ethyl simultaneously among the compounds shown by the general formula (I), it can obtain by making 1- bromo -2- fluoro ethane react under existence of the compound shown by the general formula (III) and a base.



(A and X are the same as the above among a formula.)

The quantity of the trial agent with which a reaction is presented is 2-4.5Eq of bases, and 2-4.5Eq of 1- bromo -2- fluoro ethane, and should just make these react with the reaction temperature of 20-100DEGC among a solvent to 1Eq of compounds of a general formula (III) for 1-24 hours.

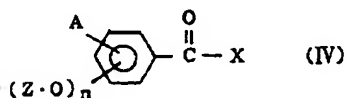
As a base,
 Sodium methoxide,
 Sodium ethoxide,
 Sodium carbonate,
 Potassium carbonate,
 Sodium hydroxide,
 Water oxidization potassium etc. can be raised.

As a solvent,
 Methanol,
 Ethanol,
 dioxane,
 N and N- dimethylform amide,
 A tetra hydro furan etc. can be raised.

After a reaction end can perform the usual post-processing, can remove water-soluble discard etc.,
 and can refine it by chromatography, distillation, etc. if needed.

(3)

The oxime compound shown by the above-mentioned general formula (II) which is the middle materials compound of this invention can make benzoyl compound and hydroxylamine salt which are shown by the general formula (IV) able to react under existence of a base, and can be obtained.



(AX and Z are the same as the above among a formula.)

To 1Eq of compounds of a general formula (IV), -- of the trial agent with which a reaction is presented are 1-1.5Eq (or sulfate) of hydroxylamine chloride salt, and 1-1.5Eq of bases, and if these are made to react with the reaction temperature of 10-70DEGC in a solvent for 1-48 hours, they can obtain an object.

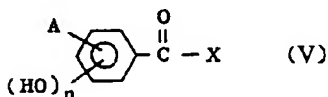
as a base -- for example
pyridine,
triethylamine,
tributylamine etc. can be raised.

As a solvent,
Methanol,
Ethanol,
dioxane,
N and N- dimethylform amide,
A tetra hydro furan etc. can be raised.

After a reaction end can perform the usual post-processing, can refine it by chromatography etc. if needed, and can obtain an object.

(4)

Benzoyl compound shown by the above-mentioned general formula (IV) which is the middle materials compound of this invention can be obtained by making hydroxybenzoyl shown by the general formula (V), dihydroxy benzoyl compound or its metal salt, and compound Z- react.



(A and X are the same as the above among a formula.)

Compound Z

(Z- is haloalkyl of C1-C4.)
dihaloalkyl of C1-C4,
haloalkenyl of C2-C4,

dihaloalkenyl of C2-C4,
halophenyl,
Poly halophenyl,
N and N- dimethylsulfamoylchloride,
N and N- dimethylcarbamoylchloride is expressed.

If an example shows compound Z-,
Iodation methyl,
Ethyl bromide
2- bromo propane,
sec- butylchloride,
1- bromo -2- fluoro ethane,
1- bromo -3- chloro -2- methyl propane,
allyl bromide,
1- chloro -2- butene,
4- bromo -1- butene,
1, 3- dichloro propene,
Iodine benzene,
2, 6- difluoro bromo benzene,
N and N- dimethylsulfamoylchloride,
N and N- dimethylcarbamoylchloride etc. can be raised.

1-1.5Eq and 2-3Eq, and a base are 1-1.5Eq and 2-3Eq similarly about compound Z', respectively,
and the quantity of the trial agent with which a reaction is presented should just make these react
with the reaction temperature of 50-L30DEGC among a solvent for 1-8 hours to hydroxybenzoyl
shown by the general formula (V) or dihydroxy benzoyl compound, and 1Eq of those metal salt.

As a base,
Sodium carbonate,
Potassium carbonate,
Sodium hydroxide,
Water oxidization potassium etc. can be raised.

as a solvent -- for example
Methanol,
Ethanol,
dioxane,
N and N- dimethylform amide,
A tetra hydro furan etc. can be raised.

After a reaction end can perform the usual post-operation, can remove water-soluble discard, and
can refine it by chromatography, distillation, a re-crystal, etc. if needed.

The harmful living thing prevention-of-the-breeding-and-extermination agent of this invention is
the purpose which can use also with the pure article of an active ingredient compound, and is used
as agricultural chemicals, and can also be used with forms, such as the form which common

agricultural chemicals can take, i.e., a water-dispersible agent, a grain agent, a powder agent, an emulsion, a water solvent, and aerosol.

When aiming at a solid agent as an additive agent and a carrier,

Soybean powder,

Vegetable powder, such as flour,

Diatomite,

Apatite,

Gypsum,

Talc,

BAIROFI light,

Mineral fine powder, such as Clay, is used.

When aiming at the drug design of a liquid,

Kerosene,

Mineral oil,

Oil,

Solvay TONAFUSA,

Xylene,

Cyclo hexane,

Cyclo hexanone,

dimethylform amide,

dimethylsulfoxide,

Alcohol,

Acetone,

Water etc. is used as a solvent.

In these tablets, if required in order to take a uniform and stable form, a surface-active agent can also be added.

Thus, the water-dispersible agent and emulsion which are obtained are diluted to concentration predetermined with water, and a powder agent and a grain agent are used by the method of sprinkling for a plant as it is as suspension liquid or an emulsion.

Moreover, the obtained tablet can also be used, mixing it with other insecticides, a tick-killing agent, a nematocidal agent, a disinfectant, a weed killer, a vegetable growth regulation agent, manure, a soil improvement agent, etc.

When using this invention compound as an active ingredient of a harmful living thing prevention-of-the-breeding-and-extermination agent, the amount of use is usually 10g per 10a. to 1000g, and the concentration for use is 10-1000 ppm.

It is 50g per 10a. to 500g preferably, and the concentration for use is 50-500 ppm.

Each of these amount of use and concentration for use changes with the kind of tablet, the time for use, the place for use, the method for use, the kind of noxious insect, damage grades, etc., and is not limited to the above-mentioned range.

(Case of the operation)

Although the example of manufacture, the example of a tablet, and the example of an examination are raised to below and this invention is explained still in detail, of course, this invention is not limited to these examples.

First, the example of manufacture of this invention compound is shown.

The example 1 of manufacture
(Manufacture of this invention compound (12))

p-allyl oxyacetophenoneoxime 2g (0.0105 mols) was dissolved in ethanol 10ml, sodium ethoxide 0.72g (0.0105 mols) and 1-bromo-2-fluoroethane 1.33g (0.0105 mols) were added to this, and mixed churning was carried out at room temperature (10-15DEGC) one whole day and night.

50ml of ethyl acetate was added to reaction liquid, 25ml of water washed twice, and the organic layer was dried with anhydrous sulfuric acid sodium.

The solvent was distilled under decompression after dryness, the residual substance was applied to thin layer chromatography (hereafter shown by T.L.C. No. [by Merck Co.]. 5717), it developed with the toluene solvent, an unreacted thing and impurities were removed, and 0.53g of mixtures of the stereoisomerism object of a compound (12) was obtained.

21.7% of yield.

In addition, a stereoisomerism object mixture may give one spot by T.L.C., and may divide it into two spots.

the physical properties, R_f value in T.L.C., and ¹H-NMR (PHX-60Si type, JEOL Co., Ltd. make) analysis value of these compounds -- the -- the [I-1 table -] -- it was shown in II-2 table.

The viscous liquid shown in the physical-properties column is the quality in the room temperature immediately after composition among front.

moreover, R_f value by T.L.C. -- the Merck Co. make -- it asked with deployment solvent toluene with the silica gel plate of No.5729.

T. That in which R_f value in L.C. has two spots described two in the parenthesis, and showed them by average value (* mark).

In addition, what calculated R_f value by deployment solvent toluene / acetone = 9 / 1 (V/V) attached and showed **.

The example 2 of manufacture

(Middle materials p-allyl oxyacetophenoneoxime composition of this invention compound (12))

p-allyl oxyacetophenone 3.52g (0.02 mols) -- ethanol 25ml -- dissolving -- chloride hydroxylamine 1.43g (0.021 mols) and triethylamine 2.02 (0.02 mols) -- in addition, overnight mixture churning was carried out at room temperature.

After having added 100ml of ethyl acetate to reaction liquid, and 50ml of water having washed twice and drying an organic layer with anhydrous sulfuric acid sodium, the solvent was distilled under decompression and the syrup-like residual substance was obtained.

The syrup-like residual substance was crystallized after that.

Weight of 3.81g, 99.7% of yield.

This compound was used in the example 1 of manufacture.

The example 3 of manufacture

(Composition of materials p-allyl oxyacetophenone of this invention compound his statement)

N/2 water oxidization potassium (ethanol solution) 73.6ml was added to p- hydroxyacetophenone 58 (0.0368 mols), and the solvent was distilled under 40 or less DEGCs and decompression.

a residual substance -- dimethylform amide 20ml, 5.08g (0.0368 mols) of potassium carbonate, and allyl bromide 4.45g (0.0368 mols) -- in addition, 60-70DEGC was made to carry out a heating reaction in oil bath

100ml of ethyl acetate was added to reaction liquid 2 hours after, 50ml of water washed twice, anhydrous sodium sulfate was added to the organic layer, drying dryness was carried out, and the solvent was distilled under decompression.

It crystallized after a while after solvent distilled.

Weight of 5.9g, 91.0% of yield.

This compound was used in the example 2 of manufacture.

The example 4 of manufacture

(Manufacture of this invention compound (22))

N/2 water oxidization potassium (ethanol solution) 300ml was added to p- hydroxy propiophenoneoxime 12.48 (0.075 mols), and the solvent was distilled under 40 or less DEGCs and decompression.

dimethylform amide 25ml was added to the residual substance, 10.4g [of potassium carbonate] (0.075 mols) and 1-bromo-2-fluoro ethane 19.1g (0.15 mols) was added after the dissolution, and 60-70DEGC was made to carry out a heating reaction in oil bath immediately.

After making it react for 2 hours, 300ml of ethyl acetate was added to reaction liquid, 150ml of water washed 3 times, anhydrous sulfuric acid sodium was added to the organic layer, and drying dryness was carried out.

After dryness, under decompression, the solvent was distilled and the rough reaction thing was obtained.

Column chromatography filled up with silica gel refined this and this 9.4g invention compound (22) was obtained.

48.7% of yield.

The example 5 of manufacture

(Composition of materials p-hydroxypropiophenoneoxime of this invention compound (22))

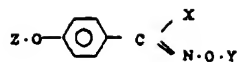
After adding ethanol 75ml to p-hydroxypropiophenone 22.53g (0.15 mols), dissolving and adding chloride hydroxylamine 10.43g (0.15 mols) and triethylamine 15.18g (0.15 mols), it was made to react by 60 in oil bath -70DEGC for 2 hours.

200ml of ethyl acetate was added after the reaction end, 100ml of water washed twice, and after drying an organic layer with anhydrous sodium sulfate, when the solvent was distilled under decompression, the 24.06g object was obtained.



97.1% of yield.



This compound was used in the example 4 of manufacture.








The I-1st tables



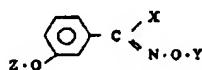
n=1, A--H

| 化合物番号 | X | Y | Z | 物性 | IR 値 | ¹ H-NMR スペクトルデータ (δ, CDCl ₃) |
|-------|--------------------------------|------------------------------------|--|-------|-----------------------------------|--|
| (1) | -H | -CH ₂ CH ₂ F | -CH ₂ CH ₂ F | 結晶性液体 | 0.48 | 3.83~4.43(m, 4H), 4.38~5.20(m, 4H), 6.87(d, 2H), 7.50(d, 2H), 8.04(s, 1H) |
| (2) | -H | -CH ₂ CH ₂ F | $\begin{matrix} \text{CH}_3 \\ \\ -\text{CH}_2\text{CHCH}_2\text{CH}_2\text{Cl} \end{matrix}$ | 同上 | 0.31 [°] (0.34, 0.28) | 1.11(d, 3H), 2.12~2.62(m, 1H), 3.62(d, 2H), 3.81(d, 2H), 4.00~4.33(m, 2H), 4.43~5.13(m, 2H), 6.86(d, 2H), 7.48(d, 2H), 8.06(s, 1H) |
| (3) | -H | -CH ₂ CH ₂ F | -CH ₂ CH=CH ₂ | 同上 | 0.75 | 3.93~4.39(m, 2H), 4.40~5.20(m, 4H), 5.07~5.58(m, 2H), 5.69~6.38(m, 1H), 6.82(d, 2H), 7.43(d, 2H), 8.01(s, 1H) |
| (4) | -H | -CH ₂ CH ₂ F |  | 同上 | 0.68 [°] (0.74, 0.61) | 3.55~4.42(m, 2H), 4.44~5.18(m, 2H), 6.60~7.30(m, 3H), 8.09(s, 1H) |
| (5) | -H | -CH ₂ CH ₂ F | $\begin{matrix} \text{CH}_3 \\ \\ -\text{SO}_2\text{N} < \text{CH}_2 \\ \\ \text{CH}_3 \end{matrix}$ | 同上 | 0.52 | 2.94(s, 6H), 4.00~4.33(m, 2H), 4.45~5.20(m, 2H), 7.21(d, 2H), 7.55(d, 2H), 8.04(s, 1H) |
| (6) | -H | -CH ₂ CH ₂ F | $\begin{matrix} \text{CH}_3 \\ \\ -\text{CN} < \text{CH}_2 \\ \\ \text{O} \end{matrix}$ | 同上 | 0.43 | 3.02(s, 6H), 3.82~4.43(m, 2H), 4.42~5.16(m, 2H), 7.00(d, 2H), 7.48(d, 2H), 7.99(s, 1H) |
| (7) | -CH ₃ | -CH ₂ CH ₂ F | -CH ₃ | 同上 | 0.51 | 2.22(s, 3H), 3.89(m, 3H), 4.02~4.40(m, 2H), 4.45~5.20(m, 2H), 6.82(d, 2H), 7.54(d, 2H) |
| (8) | -CH ₃ | -CH ₂ CH ₂ F | -C ₂ H ₅ | 同上 | 0.56 | 1.38(t, 3H), 2.22(s, 3H), 4.02(m, 2H), 4.10~4.40(m, 2H), 4.50~5.19(m, 2H), 6.84(d, 2H), 7.59(d, 2H) |
| (9) | -CH ₃ | -CH ₂ CH ₂ F | -1-C ₂ H ₅ | 同上 | 0.59 | 1.31(d, 6H), 1.88~2.30(m, 1H), 2.22(s, 3H), 3.90~4.49(m, 2H), 4.35~5.20(m, 2H), 6.84(d, 2H), 7.59(d, 2H) |
| (10) | -CH ₃ | -CH ₂ CH ₂ F | -CH ₂ CH ₂ F | 同上 | 0.57 | 2.38(m, 3H), 3.75~4.50(m, 2H), 4.00~4.40(m, 2H), 4.20~5.16(m, 2H), 4.46~5.16(m, 2H), 6.89(d, 2H), 7.61(d, 2H) |
| (11) | -CH ₃ | -CH ₂ CH ₂ F | $\begin{matrix} \text{CH}_3 \\ \\ -\text{CH}_2\text{CHCH}_2\text{CH}_2\text{Cl} \end{matrix}$ | 同上 | 0.46 [°] (0.49, 0.43) | 1.13(d, 3H), 1.81(m, 1H), 2.21(m, 3H), 3.66(d, 2H), 3.83(d, 2H), 4.10~4.32(m, 2H), 4.51~5.15(m, 2H), 6.85(d, 2H), 7.56(d, 2H) |
| (12) | -CH ₃ | -CH ₂ CH ₂ F | -CH ₂ CH=CH ₂ | 同上 | 0.66 | 2.21(s, 3H), 3.95~4.29(m, 2H), 4.40~4.62(m, 2H), 4.45~5.13(m, 2H), 5.05~5.53(m, 2H), 5.70~6.38(m, 1H), 6.82(d, 2H), 7.54(d, 2H) |
| (13) | -CH ₃ | -CH ₂ CH ₂ F | -CH ₂ CH=CHCl | 結晶性液体 | 0.69 | 2.22(s, 3H), 4.00~4.43(m, 2H), 4.35~4.63(m, 2H), 4.53~5.20(m, 2H), 5.85~6.35(m, 2H), 6.83(d, 2H), 7.62(d, 2H) |
| (14) | -CH ₃ | -CH ₂ CH ₂ F |  | 同上 | 0.77 | 1.31(t, 3H), 2.18(m, 3H), 4.22(q, 2H), 6.83~7.79(m, 3H) |
| (15) | -CH ₃ | -CH ₂ CH ₂ F |  | 同上 | 0.84 | 2.22(s, 3H), 4.15~4.33(m, 2H), 4.55~5.19(m, 2H), 6.61~7.76(m, 1H) |
| (16) | -CH ₃ | -CH ₂ CH ₂ F |  | 同上 | 0.62 | 2.26(s, 3H), 4.05~4.42(m, 2H), 4.55~5.22(m, 2H), 6.80~7.98(m, 7H) |
| (17) | -CH ₃ | -CH ₂ CH ₂ F | $\begin{matrix} \text{CH}_3 \\ \\ -\text{SO}_2\text{N} < \text{CH}_2 \\ \\ \text{CH}_3 \end{matrix}$ | 同上 | 0.67 | 2.21(s, 3H), 2.98(m, 6H), 4.00~4.42(m, 2H), 4.49~5.20(m, 2H), 7.20(d, 2H), 7.62(d, 2H) |
| (18) | -CH ₃ | -CH ₂ CH ₂ F | $\begin{matrix} \text{CH}_3 \\ \\ -\text{CN} < \text{CH}_2 \\ \\ \text{O} \end{matrix}$ | 同上 | 0.53 | 2.22(s, 3H), 3.05(m, 6H), 3.97~4.45(m, 2H), 4.44~5.20(m, 2H), 7.04(d, 2H), 7.59(d, 2H) |
| (19) | -C ₂ H ₅ | -CH ₂ CH ₂ F | -CH ₃ | 同上 | 0.53 | 1.12(t, 3H), 2.73(m, 2H), 3.78(m, 3H), 4.00~4.40(m, 2H), 4.45~5.19(m, 2H), 6.87(d, 2H), 7.55(d, 2H) |
| (20) | -C ₂ H ₅ | -CH ₂ CH ₂ F | -C ₂ H ₅ | 同上 | 0.46 | 1.12(t, 3H), 1.39(t, 2H), 2.72(m, 2H), 3.98(m, 2H), 4.05~4.37(m, 2H), 4.45~5.13(m, 2H), 6.80(d, 2H), 7.52(d, 2H) |
| (21) | -C ₂ H ₅ | -CH ₂ CH ₂ F | -1-C ₂ H ₅ | 同上 | 0.54 | 1.12(t, 3H), 1.31(d, 6H), 1.49~1.70(m, 1H), 2.76(m, 2H), 4.00~4.41(m, 2H), 4.41~5.18(m, 2H), 6.81(d, 2H), 7.52(d, 2H) |
| (22) | -C ₂ H ₅ | -CH ₂ CH ₂ F | -CH ₂ CH ₂ F | 同上 | 0.40 | 1.11(t, 3H), 2.72(m, 2H), 3.85~4.24(m, 6H), 4.92~5.21(m, 2H), 6.88(d, 2H), 7.58(d, 2H) |
| (23) | -C ₂ H ₅ | -CH ₂ CH ₂ F | -CH ₂ CH=CH ₂ | 同上 | 0.54 | 1.11(t, 3H), 2.72(m, 2H), 3.97~4.40(m, 2H), 4.42~4.73(m, 2H), 4.55~5.11(m, 2H), 5.09~5.59(m, 2H), 5.70~6.40(m, 1H), 6.87(d, 2H), 7.56(d, 2H) |
| (24) | -C ₂ H ₅ | -CH ₂ CH ₂ F | -CH ₂ CH=CHCH ₃ | 同上 | 0.58 | 1.11(t, 3H), 1.72(d, 3H), 2.71(m, 2H), 3.97~4.35(m, 2H), 4.30~4.73(m, 2H), 4.40~5.17(m, 2H), 5.35~6.05(m, 2H), 6.86(d, 2H), 7.55(d, 2H) |
| (25) | -C ₂ H ₅ | -CH ₂ CH ₂ F | -CH ₂ CH=CHCl | 同上 | 0.55 | 1.12(t, 3H), 2.75(m, 2H), 3.95~4.44(m, 2H), 4.45~4.70(m, 2H), 4.62~5.21(m, 2H), 6.05~6.35(m, 2H), 6.89(d, 2H), 7.60(d, 2H) |

| | | | | | | |
|------|---|------------------|---|-----------|------|---|
| (26) | $-C_6H_5$ | $-CH_2CH_2F$ | $-SO_2N < \begin{smallmatrix} CH_3 \\ CH_3 \end{smallmatrix}$ | 結晶性 液体 | 0.50 | 1.11(t, 3H), 2.72(q, 2H), 2.93(s, 6H), 4.09~4.37(m, 2H), 4.45~5.12(m, 2H), 7.15(d, 2H), 7.56(d, 2H) |
| (27) |  | $-CH_2CH_2F$ | $-CH_2CH_2F$ | 同上 | 0.73 | 3.80~4.75(m, 4H), 4.28~5.20(m, 4H), 6.63~7.90(m, 3H) |
| (28) |  | $-CH_2CH_2F$ | $-CH_2CH_2F$ | 同上 | 0.74 | 3.94~4.70(m, 4H), 4.25~5.16(m, 4H), 6.70~7.91(m, 3H) |
| (29) | -H | $-CH_2CH=CH_2$ | $-CH_2CH_2F$ | 同上 | 0.42 | 3.73~4.53(m, 2H), 4.19~5.20(m, 2H), 4.63(d, 2H), 4.98~5.60(m, 2H), 5.65~6.42(m, 1H), 6.74(d, 2H), 7.41(d, 2H), 7.96(s, 1H) |
| (30) | $-CH_3$ | -H | $-CH_2CH_2F$ | 同上 | 0.07 | 2.22(s, 3H), 3.90~4.58(m, 2H), 4.24~5.25(m, 2H), 6.91(d, 2H), 7.63(d, 2H), 9.58~10.17(broad, 1H) |
| (31) | $-CH_3$ | $-CH_2CH=CH_2$ | $-CH_2CH_2F$ | 同上 | 0.44 | 2.18(s, 3H), 3.78~4.49(m, 2H), 4.67(d, 2H), 4.18~5.19(m, 2H), 4.96~5.52(m, 2H), 5.62~6.45(m, 1H), 6.86(d, 2H), 7.61(d, 2H) |
| (32) | $-CH_3$ | $-CH_2COOCH_3$ | $-CH_2CH_2F$ | 同上 | 0.39 | 2.25(s, 3H), 3.73(s, 3H), 3.73~4.55(m, 2H), 4.20~5.25(m, 2H), 4.71(s, 2H), 6.88(d, 2H), 7.56(d, 2H) |
| (33) | $-C_6H_5$ | $-CH_2CH_2Cl$ | $-CH_2CH_2F$ | 同上 | 0.56 | 1.12(t, 3H), 2.76(q, 2H), 3.79(d, 2H), 3.60~4.52(m, 2H), 4.33(d, 2H), 4.21~5.23(m, 2H), 6.90(d, 2H), 7.58(d, 2H) |
| (34) | $-C_6H_5$ | $-1-C_2H_5$ | $-CH_2CH_2F$ | 同上 | 0.46 | 1.11(t, 3H), 1.91(d, 6H), 1.89~2.13(m, 1H), 2.65(q, 2H), 3.87~4.66(m, 2H), 4.13~5.21(m, 2H), 6.88(d, 2H), 5.57(d, 2H) |
| (35) | $-C_6H_5$ | $-CH_2CH=CHCH_3$ | $-CH_2CH_2F$ | 同上 | 0.53 | 1.11(t, 3H), 1.72(d, 3H), 2.73(q, 2H), 3.88~4.60(m, 2H), 4.50~4.85(m, 2H), 4.25~5.36(m, 2H), 3.63~5.92(m, 2H), 6.88(d, 2H), 7.59(d, 2H) |
| (36) | $-C_6H_5$ | $-CH_2CH=CHCl$ | $-CH_2CH_2F$ | 同上 | 0.60 | 1.11(t, 3H), 2.72(q, 2H), 3.88~4.57(m, 2H), 4.52(d, 2H), 4.25~5.28(m, 2H), 6.10~6.38(m, 2H), 6.89(d, 2H), 7.58(d, 2H) |

| | | | | | | |
|------|--|------------------|--------------|-----------|------|---|
| (37) |  | -H | $-CH_2CH_2F$ | 結晶性 液体 | 0.09 | 3.90~4.61(m, 2H), 4.25~5.23(m, 2H), 6.82~7.93(m, 3H), 9.08~9.43(broad, 1H) |
| (38) |  | $-CH_2CH=CH_2$ | $-CH_2CH_2F$ | 同上 | 0.82 | 3.80~4.51(m, 2H), 4.15~5.15(m, 2H), 4.61(d, 2H), 4.95~5.43(m, 2H), 5.62~6.60(m, 1H), 6.65~7.65(m, 3H) |
| (39) |  | $-CH_2CH=CHCH_3$ | $-CH_2CH_2F$ | 同上 | 0.86 | 1.63(d, 3H), 3.81~4.52(m, 2H), 4.59(d, 2H), 4.20~5.19(m, 2H), 5.46~5.82(m, 2H), 6.69~7.61(m, 3H) |
| (40) |  | $-CH_2CH=CHCl$ | $-CH_2CH_2F$ | 同上 | 0.84 | 3.75~4.50(m, 2H), 4.60(q, 2H), 4.20~5.20(m, 2H), 6.01~6.22(m, 2H), 6.55~7.13(m, 3H) |
| (41) |  | -H | $-CH_2CH_2F$ | 同上 | 0.09 | 3.89~4.53(m, 2H), 4.22~5.21(m, 2H), 6.79~7.93(m, 3H), 8.90~9.21(broad, 1H) |
| (42) |  | $-CH_2CH=CH_2$ | $-CH_2CH_2F$ | 同上 | 0.83 | 3.85~4.55(m, 2H), 4.65(d, 2H), 4.21~5.16(m, 2H), 5.00~5.42(m, 2H), 5.68~6.12(m, 1H), 6.72~7.60(m, 3H) |
| (43) |  | $-CH_2CH=CHCl$ | $-CH_2CH_2F$ | 同上 | 0.85 | 3.85~4.52(m, 2H), 4.61(d, 2H), 4.22~5.20(m, 2H), 6.04~6.22(m, 2H), 6.72~7.58(m, 3H) |

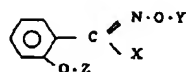
The I-2nd tables



| 化合物 番号 | X | Y | Z | 物性 | γ_L/γ_H | 1H -NMR スペクトルデータ (δ , $CDCl_3$) |
|-----------|-----------|----------------|---|-----------|---------------------|---|
| (44) | -H | $-CH_2CH_2F$ | $-1-C_6H_5$ | 結晶性 液体 | 0.51 | 1.32(d, 6H), 1.55~1.80(m, 1H), 4.05~4.80(m, 2H), 4.43~5.19(m, 2H), 6.75~7.50(m, 4H), 8.08(s, 1H) |
| (45) | -H | $-CH_2CH_2F$ | $-CH_2CH_2F$ | 同上 | 0.47 | 3.83~4.72(m, 2H), 4.10~5.26(m, 6H), 6.75~7.35(m, 4H), 8.03(s, 1H) |
| (46) | -H | $-CH_2CH_2F$ | $-CH_2CH=CH_2$ | 同上 | 0.50 | 4.05~4.80(m, 2H), 4.46~4.75(m, 2H), 4.45~5.12(m, 2H), 5.15~5.64(m, 1H), 6.80~7.55(m, 4H), 8.10(s, 1H) |
| (47) | $-CH_3$ | $-CH_2CH_2F$ | $-1-C_6H_5$ | 同上 | 0.36 | 1.31(d, 6H), 1.40~1.55(m, 1H), 2.20(s, 3H), 4.04~4.70(m, 2H), 4.40~5.15(m, 2H), 6.96~7.27(m, 4H) |
| (48) | $-CH_3$ | $-CH_2CH_2F$ | $-CH_2CH_2F$ | 同上 | 0.36 | 2.22(s, 3H), 3.85~4.76(m, 2H), 4.13~5.24(m, 6H), 6.75~7.45(m, 4H) |
| (49) | $-CH_3$ | $-CH_2CH_2F$ | $-CH_2CH=CH_2$ | 同上 | 0.47 | 2.21(s, 3H), 4.15~4.80(m, 2H), 4.60(d, 2H), 4.45~5.14(m, 2H), 4.91~5.57(m, 2H), 5.70~6.30(m, 1H), 6.65~7.40(m, 4H) |
| (50) | $-CH_3$ | $-CH_2CH_2F$ | $\begin{smallmatrix} CH_3 \\ O \\ CH_3 \end{smallmatrix}$ | 同上 | 0.16 | 2.20(s, 3H), 3.01(s, 6H), 3.90~4.62(m, 2H), 4.40~5.10(m, 2H), 6.80~7.35(m, 4H) |
| (51) | $-C_6H_5$ | $-CH_2CH_2F$ | $-CH_2CH_2F$ | 同上 | 0.44 | 1.12(t, 3H), 2.77(q, 2H), 3.89~4.80(m, 2H), 4.18~5.30(m, 6H), 6.75~7.35(m, 4H) |
| (52) | $-C_6H_5$ | $-CH_2CH_2F$ | $-CH_2CH=CH_2$ | 同上 | 0.55 | 1.13(t, 3H), 2.76(q, 2H), 4.05~4.80(m, 2H), 4.62(d, 2H), 4.46~5.30(m, 2H), 5.10~5.65(m, 2H), 5.75~6.30(m, 1H), 6.65~7.60(m, 4H) |
| (53) | $-C_6H_5$ | $-CH_2CH_2F$ | $-CH_2CH=CHCl$ | 同上 | 0.57 | 1.12(t, 3H), 2.76(q, 2H), 3.90~4.85(m, 2H), 4.52(d, 2H), 4.50~5.19(m, 2H), 5.83~6.40(m, 2H), 6.80~7.50(m, 4H) |
| (54) | $-C_6H_5$ | $-CH_2CH_2F$ | $-CH_2CH=CHCH_3$ | 同上 | 0.60 | 1.12(t, 3H), 1.73(d, 2H), 2.77(q, 2H), 4.05~4.90(m, 2H), 4.40~4.63(m, 2H), 4.56~5.20(m, 2H), 5.70~5.90(m, 2H), 6.75~7.40(m, 4H) |
| (55) | -H | -H | $-CH_2CH_2F$ | 同上 | 0.11 | 3.98~4.75(m, 2H), 4.50~5.18(m, 2H), 6.70~7.45(m, 4H), 8.01(s, 1H), 8.75~9.11(broad, 1H) |
| (56) | -H | $-CH_2CH=CH_2$ | $-CH_2CH_2F$ | 同上 | 0.66 | 3.80~4.56(m, 2H), 4.67(d, 2H), 4.25~5.24(m, 2H), 5.10~5.60(m, 2H), 5.65~6.35(m, 1H), 6.78~7.50(m, 4H), 8.07(s, 1H) |

| | | | | | | |
|------|--------------------------------|---------------------------------------|------------------------------------|-------|------|---|
| (57) | -H | -CH ₂ CH=CHCH ₃ | -CH ₂ CH ₂ F | 脂肪性液体 | 0.64 | 1.74(d, 2H), 3.83~4.75(m, 2H), 4.25~4.55(m, 2H), 4.17~5.18(m, 2H), 5.57~5.85(m, 2H), 6.65~7.40(m, 4H), 7.91(s, 1H) |
| (58) | -H | -CH ₂ CH=CHCl | -CH ₂ CH ₂ F | 同上 | 0.69 | 3.89~4.54(m, 2H), 4.63(d, 2H), 4.25~5.23(m, 2H), 6.09~6.35(m, 2H), 6.70~7.50(m, 4H), 8.02(s, 1H) |
| (59) | -CH ₃ | -H | -CH ₂ CH ₂ F | 同上 | 0.09 | 2.14(s, 3H), 3.82~4.55(m, 2H), 4.20~5.19(m, 2H), 6.74~7.32(m, 4H) |
| (60) | -CH ₃ | -CH ₂ CH=CH ₂ | -CH ₂ CH ₂ F | 同上 | 0.38 | 2.18(s, 3H), 3.80~4.50(m, 2H), 4.29~5.17(m, 2H), 4.63(d, 2H), 5.05~5.52(m, 2H), 5.68~6.49(m, 1H), 6.70~7.36(m, 4H) |
| (61) | -CH ₃ | -CH ₂ CH=CHCH ₃ | -CH ₂ CH ₂ F | 同上 | 0.52 | 1.72(d, 3H), 2.18(s, 1H), 3.74~4.82(m, 2H), 4.55~4.80(m, 2H), 4.23~5.18(m, 2H), 5.61~6.00(m, 2H), 6.74~7.60(m, 4H) |
| (62) | -CH ₃ | -CH ₂ CH=CHCl | -CH ₂ CH ₂ F | 同上 | 0.40 | 2.19(s, 3H), 3.86~4.55(m, 2H), 4.20~5.24(m, 2H), 4.55~4.78(m, 2H), 6.02~6.37(m, 2H), 6.70~7.60(m, 4H) |
| (63) | -C ₂ H ₅ | -H | -CH ₂ CH ₂ F | 同上 | 0.08 | 1.11(t, 3H), 2.74(q, 2H), 3.86~4.57(m, 2H), 4.17~5.19(m, 2H), 6.80~7.58(m, 4H), 8.80~9.21(broad, 1H) |
| (64) | -C ₂ H ₅ | -CH ₂ CH=CH ₂ | -CH ₂ CH ₂ F | 同上 | 0.52 | 1.12(t, 3H), 2.75(q, 2H), 3.82~4.60(m, 2H), 4.15~5.22(m, 2H), 4.68(d, 2H), 5.00~5.52(m, 2H), 5.65~6.45(m, 1H), 6.74~7.60(m, 4H) |
| (65) | -C ₂ H ₅ | -CH ₂ CH=CHCH ₃ | -CH ₂ CH ₂ F | 同上 | 0.53 | 1.10(t, 3H), 1.72(d, 3H), 2.73(q, 2H), 3.88~4.51(m, 2H), 4.62(d, 2H), 4.20~5.30(m, 2H), 5.62~6.10(m, 2H), 6.74~7.60(m, 4H) |
| (66) | -C ₂ H ₅ | -CH ₂ CH=CHCl | -CH ₂ CH ₂ F | 同上 | 0.61 | 1.11(t, 3H), 2.71(q, 2H), 3.88~4.55(m, 2H), 4.62(d, 2H), 4.19~5.25(m, 2H), 6.07~6.35(m, 2H), 6.55~7.45(m, 4H) |

The I-3rd tables



n = 1, A = -H

| 化合物 | X | Y | Z | 物性 | IR値 | ¹ H-NMR スペクトルデータ (δ, CDCl ₃) |
|------|--------------------------------|---------------------------------------|---------------------------------------|-------|------|---|
| (67) | -H | -CH ₂ CH ₂ F | -i-C ₂ H ₅ | 脂肪性液体 | 0.70 | 1.31(d, 6H), 3.99~4.38(q, 1H), 4.07~4.77(m, 2H), 4.40~5.17(m, 2H), 6.69~7.91(m, 4H), 8.54(s, 1H) |
| (68) | -H | -CH ₂ CH ₂ F | -CH ₂ CH ₂ F | 同上 | 0.62 | 3.80~5.15(m, 8H), 6.65~7.83(m, 4H), 8.49(s, 1H) |
| (69) | -H | -CH ₂ CH ₂ F | -CH ₂ CH=CH ₂ | 同上 | 0.71 | 4.00~4.75(m, 2H), 4.40~4.80(m, 2H), 4.40~5.25(m, 2H), 4.92~5.60(m, 2H), 5.70~6.40(m, 1H), 6.70~8.00(m, 2H), 6.68~7.95(m, 4H), 8.50(s, 1H) |
| (70) | -H | -CH ₂ CH ₂ F | -CH ₂ CH=CHCl | 同上 | 0.72 | 4.00~4.88(m, 2H), 4.47(d, 2H), 4.53~5.17(m, 2H), 5.80~6.26(m, 2H), 6.68~7.95(m, 4H), 8.50(s, 1H) |
| (71) | -H | -CH ₂ CH ₂ F | -CH ₂ CH=CHCH ₃ | 同上 | 0.71 | 1.68(d, 3H), 4.00~4.80(m, 2H), 4.44(d, 2H), 4.31~5.20(m, 2H), 5.35~5.90(m, 2H), 6.70~7.96(m, 4H), 8.59(s, 1H) |
| (72) | -CH ₃ | -CH ₂ CH ₂ F | -CH ₂ CH ₂ F | 同上 | 0.45 | 2.24(s, 3H), 3.82~4.70(m, 6H), 4.91~5.16(m, 2H), 6.70~7.48(m, 4H) |
| (73) | -C ₂ H ₅ | -CH ₂ CH ₂ F | -CH ₂ CH ₂ F | 同上 | 0.40 | 1.09(t, 3H), 2.31(q, 2H), 3.79~4.70(m, 6H), 4.80~5.20(m, 2H), 6.72~7.78(m, 4H) |
| (74) | -H | -H | -CH ₂ CH ₂ F | 同上 | 0.11 | 3.70~4.63(m, 2H), 4.16~5.19(m, 2H), 6.80~7.76(m, 4H), 8.50~8.91(broad, 1H) |
| (75) | -H | -i-C ₂ H ₅ | -CH ₂ CH ₂ F | 同上 | 0.69 | 1.80(d, 6H), 3.83~4.52(m, 2H), 4.22~4.68(m, 1H), 4.22~5.21(m, 2H), 6.70~7.95(m, 4H), 8.45(s, 1H) |
| (76) | -H | -CH ₂ CH ₂ Cl | -CH ₂ CH ₂ F | 同上 | 0.72 | 3.60~4.52(m, 6H), 4.20~5.22(m, 2H), 6.71~7.91(m, 4H), 8.52(s, 1H) |
| (77) | -H | -CH ₂ CH=CH ₂ | -CH ₂ CH ₂ F | 同上 | 0.58 | 3.80~4.51(m, 2H), 4.65(d, 2H), 4.19~5.22(m, 2H), 5.18~5.55(m, 2H), 5.70~6.45(m, 1H), 6.70~8.00(m, 4H), 8.53(s, 1H) |
| (78) | -H | -CH ₂ CH=CHCl | -CH ₂ CH ₂ F | 同上 | 0.65 | 3.90~4.51(m, 2H), 4.68(d, 2H), 4.30~5.25(m, 2H), 5.90~6.34(m, 2H), 6.78~7.98(m, 4H), 8.56(s, 1H) |
| (79) | -H | -CH ₂ CH=CHCH ₃ | -CH ₂ CH ₂ F | 同上 | 0.59 | 1.73(d, 3H), 3.85~4.50(m, 2H), 4.20~4.85(m, 2H), 4.20~5.22(m, 2H), 5.60~5.96(m, 2H), 6.73~8.00(m, 4H), 8.58(s, 1H) |

| | | | | | | |
|------|--------------------------------|---------------------------------------|------------------------------------|-----------|------|---|
| (80) | -CH ₃ | -H | -CH ₂ CH ₂ F | 防錆性 液体 | 0.13 | 2.19(s,3H), 3.85~4.53(m,2H), 4.23~5.18(m,2H), 6.72~7.45(m,4H), 9.03~9.15(broad,1H) |
| (81) | -CH ₃ | -i-C ₃ H ₇ | -CH ₂ CH ₂ F | 同上 | 0.44 | 1.29(d,6H), 2.20(s,3H), 3.85~4.48(m,2H), 4.15~4.65(m,1H), 4.20~5.15(m,2H), 6.72~7.48(m,4H) |
| (82) | -CH ₃ | -CH ₂ CH ₂ Cl | -CH ₂ CH ₂ F | 同上 | 0.52 | 2.21(s,3H), 3.60~4.53(m,6H), 4.15~5.20(m,2H), 6.74~7.50(m,4H) |
| (83) | -CH ₃ | -CH ₂ CH=CH ₂ | -CH ₂ CH ₂ F | 同上 | 0.48 | 2.22(s,3H), 3.83~4.55(m,2H), 4.57(d,2H), 4.15~5.20(m,2H), 4.98~5.13(m,2H), 3.85~6.43(m,1H), 6.72~7.48(m,4H) |
| (84) | -CH ₃ | -CH ₂ CH=CHCl | -CH ₂ CH ₂ F | 同上 | 0.53 | 2.21(s,3H), 3.80~4.50(m,2H), 4.62(d,2H), 4.15~5.14(m,2H), 6.03~6.30(m,2H), 6.70~7.44(m,4H) |
| (85) | -CH ₃ | -CH ₂ CH=CHCH ₃ | -CH ₂ CH ₂ F | 同上 | 0.49 | 1.22(d,3H), 2.22(s,3H), 3.80~4.53(m,2H), 4.15~5.17(m,2H), 5.53~5.73(m,2H), 3.63~5.92(m,2H), 6.73~7.50(m,4H) |
| (86) | -C ₆ H ₅ | -H | -CH ₂ CH ₂ F | 同上 | 0.13 | 1.05(t,3H), 2.78(q,2H), 3.78~4.55(m,2H), 4.15~5.22(m,2H), 6.85~7.56(m,4H), 8.83~9.05(broad,1H) |
| (87) | -C ₆ H ₅ | -CH ₂ CH ₂ Cl | -CH ₂ CH ₂ F | 同上 | 0.37 | 1.02(t,3H), 2.75(q,2H), 3.62~4.60(m,6H), 4.13~5.15(m,2H), 6.83~7.54(m,4H) |
| (88) | -C ₆ H ₅ | -CH ₂ CH=CH ₂ | -CH ₂ CH ₂ F | 同上 | 0.36 | 1.09(t,3H), 2.90(q,2H), 3.85~4.55(m,2H), 4.15~5.24(m,2H), 4.44(d,2H), 4.95~5.50(m,2H), 5.75~6.42(m,1H), 6.85~7.83(m,4H) |
| (89) | -C ₆ H ₅ | -CH ₂ CH=CHCl | -CH ₂ CH ₂ F | 同上 | 0.51 | 1.02(t,3H), 2.70(q,2H), 3.83~4.54(m,2H), 4.16~5.17(m,2H), 4.60(d,2H), 6.03~6.29(m,2H), 6.72~7.48(m,4H) |
| (90) | -C ₆ H ₅ | -CH ₂ CH=CHCH ₃ | -CH ₂ CH ₂ F | 同上 | 0.45 | 1.03(t,3H), 1.74(d,3H), 2.76(q,2H), 3.83~4.50(m,2H), 4.14~5.20(m,2H), 4.35~4.82(m,2H), 5.56~5.90(m,2H), 6.65~7.50(m,4H) |

The I-4th tables

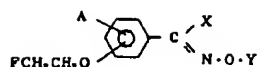


** 展開溶媒 トルエン/アセトン=9/1

| 化合物 | A | X | -OZ | 物性 | T _L C R _F 値 | ¹ H-NMR スペクトルデータ (δ, CDCl ₃) |
|-------|----------------------------------|------------------|---|-----------|--------------------------------------|--|
| (91) | 2-OH | | 4-OCH ₂ CH ₂ F | 防錆性 液体 | 0.23 | 3.80~4.68(m,4H), 4.15~5.26(m,4H), 6.43~7.75(m,8H), 8.30~8.52(broad,1H) |
| (92) | 3-OCH ₃ | -H | 4-OCH ₂ CH ₂ F | 同上 | 0.39 | 3.82(s,3H), 3.65~4.20(m,4H), 4.05~5.21(m,4H), 6.65~7.27(m,3H), 7.98(s,1H) |
| (93) | 3-OCH ₃ | -CH ₃ | 4-OCH ₂ CH ₂ F | 無色 固体 | 0.33 | 2.21(s,3H), 3.65~4.40(m,4H), 3.83(s,3H), 4.35~5.22(m,4H), 6.69~7.32(m,3H) |
| (94) | 3-OCH ₃ | -H | 2-OCH ₂ CH ₂ F | 防錆性 液体 | 0.26 | 3.80(s,3H), 3.85~4.70(m,4H), 4.38~5.13(m,4H), 6.72~7.49(m,3H), 8.52(s,1H) |
| (95) | 4-OCH ₃ | -H | 3-OCH ₂ CH ₂ F | 同上 | 0.27 | 3.84(s,3H), 3.74~4.56(m,4H), 4.27~5.26(m,4H), 6.75~7.41(m,3H), 7.99(s,1H) |
| (96) | 4-OCH ₃ | -H | 2-OCH ₂ CH ₂ F | 同上 | 0.26 | 3.81(s,3H), 3.67~4.48(m,4H), 4.33~5.24(m,4H), 6.69~7.39(m,3H), 8.03(s,1H) |
| (97) | 3-OC ₂ H ₅ | -H | 4-OCH ₂ CH ₂ F | 無色 固体 | 0.72 ** | 1.31(d,6H), 1.42(t,3H), 1.78~2.22(m,1H), 4.04(q,2H), 3.95~4.15(m,2H), 4.41~5.09(m,2H), 6.50~7.20(m,3H), 7.92(s,1H) |
| (98) | 3-OCH ₃ | -H | 4-OCH ₂ CH=CH ₂ | 防錆性 液体 | 0.79 ** | 3.81(s,3H), 3.99~4.65(m,2H), 4.52(d,2H), 4.45~5.10(m,2H), 5.05~5.55(m,2H), 5.70~6.40(m,1H), 6.88~7.24(m,3H), 8.00(s,1H) |
| (99) | 3-OCH ₃ | -CH ₃ | 4-OCH ₂ CH=CH ₂ | 無色 固体 | 0.42 ** | 2.20(s,3H), 3.88(s,3H), 4.05~4.75(m,2H), 4.60(d,2H), 4.51~5.29(m,2H), 5.11~5.59(m,2H), 5.88~6.42(m,1H), 6.71~7.39(m,3H) |
| (100) | 3-OCH ₃ | -H | 4-OCH ₂ CH=CHCH ₃ | 同上 | 0.73 ** | 1.70(d,3H), 3.81(s,3H), 3.97~4.70(m,2H), 4.40~5.13(m,2H), 4.50~4.71(m,2H), 5.60~5.90(m,2H), 6.63~7.21(m,3H), 8.00(s,1H) |

| | | | | | | |
|-------|--------------------|------------------|---|-----------|--------|--|
| (101) | 3-OCH ₃ | -CH ₃ | 4-OCH ₂ CH=CHCH ₃ | 無色 固体 | 0.76** | 1.71(d,3H), 2.23(s,3H), 3.89(s,3H), 4.03~4.65(m,2H), 4.41~5.20(m,2H), 4.50~4.85(m,2H), 5.65~6.00(m,2H), 6.70~7.40(m,3H) |
| (102) | 4-OCH ₃ | -H | 2-OCH ₂ CH=CH ₂ | 防錆性 液体 | 0.43 | 3.79(s,3H), 3.66~4.46(m,2H), 4.50(d,2H), 4.25~5.21(m,2H), 5.67~6.36(m,1H), 6.83~7.23(m,3H), 8.12(s,1H) |
| (103) | 3-Cl | -H | 4-OCH ₂ CH=CHCl | 同上 | 0.51 | 4.05~4.73(m,2H), 4.58(d,1H), 4.83(d,1H), 4.50~5.17(m,2H), 6.04~6.42(m,2H), 6.72~7.70(m,3H), 7.99(s,1H) |
| (104) | 3-OCH ₃ | -H | 4-OCH ₂ CH=CHCl | 無色 固体 | 0.81** | 3.81(s,3H), 3.96~4.65(m,2H), 4.50(d,1H), 4.76(d,1H), 4.40~5.11(m,2H), 5.80~6.21(m,2H), 6.40~7.21(m,3H), 7.98(s,1H) |
| (105) | 3-OCH ₃ | -CH ₃ | 4-OCH ₂ CH=CHCl | 防錆性 液体 | 0.83** | 2.21(s,3H), 3.83(s,3H), 4.00~4.69(m,2H), 4.51(d,1H), 4.79(d,1H), 4.42~5.13(m,2H), 5.82~6.31(m,2H), 6.63~7.32(m,3H) |
| (106) | 4-OCH ₃ | -H | 2-OCH ₂ CH=CHCl | 同上 | 0.52 | 3.82(s,3H), 3.76~4.41(m,2H), 4.52(d,1H), 4.81(d,1H), 4.45~5.21(m,2H), 5.78~6.29(m,2H), 6.57~7.29(m,3H), 8.09(s,1H) |
| (107) | 3-Cl | -H | 4-OCH ₂ CH ₂ F | 同上 | 0.40 | 3.90~4.73(m,4H), 4.25~5.21(m,4H), 6.73~7.66(m,3H), 7.97(s,1H) |
| (108) | 3-Cl | -H | 4-OCH ₂ CH=CH ₂ | 同上 | 0.54 | 3.95~4.65(m,2H), 4.45~4.61(m,2H), 4.45~5.05(m,2H), 5.05~5.50(m,2H), 5.76~6.35(m,1H), 6.08~7.63(m,3H), 7.93(s,1H) |

The I-5th tables

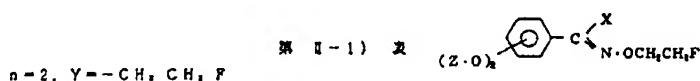
n = 1, Z = -CH₂CH₂F

** 展開溶媒 トルエン/アセトン=9/1

| 化合物 | A | X | Y | の位置 | 物性 | T.L.C Rf値 | ¹ H-NMR スペクトルデータ (δ, CDCI ₃) |
|-------|--------------------|------------------|---------------------------------------|-----|-----------|-----------|---|
| (109) | 3-Cl | -H | -CH< CH ₃ | 4位 | 防錆性 液体 | 0.67 | 1.28(d,6H), 1.80~2.35(m,1H), 3.85~4.58(m,2H), 4.15~5.23(m,2H), 6.73~7.70(m,3H), 7.90(s,1H) |
| (110) | 3-OCH ₃ | -H | -CH ₂ CH-CHCl | 4位 | 無色 固体 | 0.82 ** | 3.81(s,3H), 3.60~4.50(m,2H), 4.55(d,1H), 4.80(d,1H), 4.20~5.19(m,2H), 6.03~6.24(m,2H), 6.60~7.21(m,3H), 7.92(s,1H) |
| (111) | 3-OCH ₃ | -H | -CH ₂ CH-CHCH ₃ | 4位 | 同上 | 0.82 ** | 1.72(d,3H), 3.89(s,3H), 3.70~4.53(m,2H), 4.40~4.80(m,2H), 4.15~5.19(m,2H), 5.50~5.85(m,2H), 6.60~7.28(m,3H), 7.93(s,1H) |
| (112) | 3-OCH ₃ | -H | -H | 4位 | 同上 | 0.15 ** | 3.83(s,3H), 3.82~4.53(m,2H), 4.24~5.26(m,2H), 6.73~7.31(m,3H), 7.96(s,1H), 8.53~8.87(broad,1H) |
| (113) | 3-OCH ₃ | -CH ₃ | -H | 4位 | 同上 | 0.16 ** | 2.34(s,3H), 3.73~4.55(m,2H), 3.79(s,3H), 4.16~5.18(m,2H), 6.81~7.54(m,3H) |
| (114) | 3-OCH ₃ | -H | -CH< CH ₃ | 4位 | 同上 | 0.83 ** | 1.28(d,6H), 1.80~2.05(m,1H), 3.99(s,3H), 3.85~4.60(m,2H), 4.24~5.30(m,2H), 6.85~7.44(m,3H), 7.95(s,1H) |
| (115) | 3-OCH ₃ | -H | -sec C ₂ H ₅ | 4位 | 同上 | 0.85 ** | 0.93(s,3H), 1.27(d,3H), 1.50~1.60(m,1H), 1.55~1.99(m,2H), 3.91(s,3H), 3.86~4.61(m,2H), 4.30~5.27(m,2H), 6.83~7.34(m,3H), 7.98(s,1H) |
| (116) | 4-OCH ₃ | -H | -H | 3位 | 同上 | 0.07 | 3.89(s,3H), 3.81~4.46(m,2H), 4.20~5.24(m,2H), 6.84~7.36(m,3H), 7.93(s,1H), 8.74~8.83(broad,1H) |
| (117) | 3-OCH ₃ | -CH ₃ | -CH< CH ₃ | 4位 | 無色 固体 | 0.85 ** | 1.30(d,6H), 1.75~1.86(m,1H), 2.19(m,3H), 3.90(s,3H), 3.85~4.60(m,2H), 4.21~5.25(m,2H), 6.75~7.34(m,3H) |
| (118) | 3-OCH ₃ | -CH ₃ | -sec C ₂ H ₅ | 4位 | 防錆性 液体 | 0.93 ** | 0.94(s,3H), 1.29(d,3H), 1.35~2.00(m,3H), 2.20(s,3H), 3.88(s,3H), 3.84~4.65(m,2H), 4.30~5.35(m,2H), 6.75~7.42(m,3H) |

| | | | | | | | |
|-------|--------------------|------------------|---|----|-----------|---------|--|
| (119) | 3-OCH ₃ | -CH ₃ | -CH ₂ CH-CH ₂ | 4位 | 防錆性 液体 | 0.87 ** | 2.20(s,3H), 2.50(s,2H), 3.85~4.65(m,2H), 3.90(s,3H), 4.20~4.45(m,2H), 4.30~5.30(m,2H), 4.40~5.28(m,2H), 5.55~6.20(m,1H), 6.83~7.45(m,3H) |
| (120) | 4-OCH ₃ | -H | -CH< CH ₃ | 3位 | 同上 | 0.34 | 1.30(d,6H), 1.83~2.01(m,1H), 3.89(s,3H), 3.81~4.59(m,2H), 4.29~5.25(m,2H), 6.67~7.40(m,3H), 8.51(s,1H) |
| (121) | 4-OCH ₃ | -H | -CH ₂ CH ₂ Cl | 3位 | 同上 | 0.37 | 3.90(s,3H), 3.78~4.56(m,6H), 4.17~5.23(m,2H), 6.65~7.41(m,3H), 8.49(s,1H) |
| (122) | 4-OCH ₃ | -H | -CH ₂ CH ₂ Cl | 2位 | 同上 | 0.37 | 3.88(s,3H), 3.81~4.49(m,6H), 4.20~5.31(m,2H), 6.71~7.39(m,3H), 8.31(s,1H) |
| (123) | 3-OCH ₃ | -CH ₃ | -CH ₂ CH-CHCH ₃ | 4位 | 無色 固体 | 0.86 ** | 1.73(d,3H), 2.20(s,3H), 3.84(s,3H), 3.84~4.52(m,2H), 4.24~5.23(m,2H), 4.52~4.85(m,2H), 5.53~5.90(m,2H), 6.72~7.50(m,3H) |
| (124) | 4-OCH ₃ | -H | -CH ₂ CH-CH ₂ | 3位 | 防錆性 液体 | 0.36 | 3.81(s,3H), 3.77~4.58(m,2H), 4.52(d,2H), 4.20~5.19(m,2H), 5.07~5.53(m,2H), 3.76~6.38(m,1H), 6.74~7.48(m,3H), 8.49(s,1H) |
| (125) | 3-OCH ₃ | -CH ₃ | -CH ₂ CH-CHCl | 4位 | 無色 固体 | 0.86 ** | 2.18(s,3H), 3.65~4.55(m,2H), 3.85(s,3H), 4.20~5.21(m,2H), 4.52(d,1H), 4.80~5.20(m,1H), 6.09~6.31(m,2H), 6.70~7.52(m,3H) |
| (126) | 3-OCH ₃ | -H | -CH ₂ CH ₂ CH-CH ₂ | 2位 | 防錆性 液体 | 0.45 | 2.45(s,3H), 3.82(s,3H), 3.88~4.62(m,2H), 4.16~5.10(m,2H), 4.41~4.63(m,1H), 3.10~5.32(m,2H), 5.57~6.23(m,2H), 6.87~7.60(m,3H), 8.50(s,1H) |
| (127) | 4-OCH ₃ | -H | -CH ₂ CH-CHCl | 3位 | 同上 | 0.47 | 3.89(s,3H), 3.66~4.51(m,2H), 4.53(d,1H), 5.08~5.47(m,2H), 4.51(d,1H), 4.83(d,1H), 5.76~6.22(m,2H), 6.73~7.58(m,3H), 8.49(s,1H) |
| (128) | 3-Cl | -H | -CH ₂ CH-CHCl | 4位 | 防錆性 液体 | 0.69 | 3.89~4.53(m,2H), 4.25~5.30(m,2H), 4.61(d,1H), 4.87(d,1H), 6.07~6.31(m,2H), 6.80~7.72(m,3H), 7.96(s,1H) |
| (129) | 3-Cl | -H | -H | 4位 | 無色 固体 | 0.07 | 3.85~4.47(m,2H), 4.18~5.16(m,2H), 6.82~7.69(m,3H), 8.01(s,1H), 9.14~9.43(broad,1H) |
| (130) | 3-Cl | -H | -CH ₂ CH-CH ₂ | 4位 | 防錆性 液体 | 0.64 | 3.90~4.55(m,2H), 4.63(m,2H), 4.25~5.20(m,2H), 5.01~5.50(m,2H), 5.70~6.40(m,1H), 6.73~7.70(m,3H), 7.95(s,1H) |

the -- II-1 table

n = 2, Y = -CH₂CH₂F

** 展開溶媒 トルエン/アセトン=9/1


| 化合物 | A | X | (OZ) ₂ の位置 | 物性 | T.L.C Rf値 | ¹ H-NMR スペクトルデータ (δ, CDCI ₃) |
|-------|----|------------------|--|-----------|-----------|--|
| (131) | -H | | 2,4-(OCH ₂ CH ₂ F) ₂ | 防錆性 液体 | 0.36 | 3.80~4.68(m,6H), 4.10~5.20(m,6H), 6.43~7.57(m,8H) |
| (132) | -H | -CH ₃ | 3,4-(OCH ₂ CH ₂ CH ₃) ₂ | 同上 | 0.77 ** | 2.19(s,3H), 3.95~4.70(m,2H), 4.45~4.70(m,2H), 4.45~5.10(m,2H), 5.05~5.53(m,2H), 5.69~6.30(m,1H), 6.65~7.25(m,3H) |
| (133) | -H | -CH ₃ | 2,4-(OCH ₂ CH ₂ CH ₃) ₂ | 同上 | 0.79 ** | 2.27(s,3H), 3.80~4.75(m,2H), 4.40~4.55(m,2H), 4.40~5.11(m,2H), 5.05~5.52(m,2H), 5.15~6.12(m,1H), 6.36~7.50(m,3H) |

the -- II-2 table

$$\text{FCH}_2\text{CH}_2\text{O}-\text{C}_6\text{H}_4-\text{C}(\text{N}(\text{O} \cdot \text{Y}))(\text{X})-\text{OCH}_2\text{CH}_2\text{F}$$

$n = 2, Z = -\text{CH}_2\text{CH}_2\text{F}$

第 II - 2) 表

| 化合物番号 | A | X | Y | 物性 | P.L.C. 値 | ¹ H-NMR スペクトルデータ (δ, CDCl ₃) |
|-------|----|---|---------------------------------------|------|----------|--|
| (134) | -H |  | -H | 無色固体 | 0.28 ** | 3.88~4.65(m, 4H), 4.32~5.21(m, 4H), 6.55~7.34(m, 8H) 8.75~8.98(broad, 1H) |
| (135) | -H | -CH ₃ | -CH ₂ CH ₂ F | 粘性液体 | 0.73 ** | 2.20(s, 3H), 3.78~4.50(m, 6H), 4.05~5.15(m, 6H), 6.30~7.33(m, 3H) |
| (136) | -H | -CH ₃ | -CH ₂ CH ₂ CHCl | 同上 | 0.79 ** | 2.27(s, 3H), 3.82~4.50(m, 4H), 4.20~5.20(m, 4H), 4.18(d, 1H), 4.82(d, 1H), 5.83~6.28(m, 2H), 6.30~7.40(m, 3H) |
| (137) | -H | -CH ₃ | -CH ₂ COO·CH ₃ | 同上 | 0.30 | 2.25(s, 3H), 3.84(s, 3H), 3.80~4.58(m, 4H), 4.20~5.21(m, 4H), 4.68(s, 2H), 6.30~7.40(m, 3H) |

Next, the example of a tablet is shown.

in addition, a compound number -- the -- the [I-1 table -] -- this invention compound number of II-2 table shows.

The example 1 of a tablet

Emulsion

20 weight parts, the Tween 20 (product made from Wako Pure Chem side) 20 weight part, and the xylene 60 weight part were enough mixed for this invention compound (1) or (29), and the emulsion was obtained 20%.

The example 2 of a tablet

water-dispersible agent

25 weight parts, the diatomite 65 weight part, the higher alcohol sulfate ester 5 weight part, and the alkylnaphthalene sulfonic acid 5 weight part were mixed uniformly, this invention compound (1) or (29) were ground minutely, and 25% of water-dispersible agent was obtained.

The example 3 of a tablet

Particle agent

After having improved 3 weight parts, the talc 45 weight part, the vent night 45 weight part, the lignin-sulfonic-acid sodium 5 weight part, and the dodecylbenzenesulfonic acid sodium 2 weight part pulverization mixture of this invention compound (1) or (29), adding water and arranging matters, granulation dryness was carried out so that it might become 30-300 microns of particle diameter, and 3% of particle agent was obtained.

The following examples of an examination show that the compound by this invention is useful as a harmful living thing prevention-of-the-breeding-and-extermination agent.

in addition, a mandarin orange red spider and the example of an examination of a diamondback moth -- the average value of 2 repetition -- a planthopper and a lotus -- the average value of 3 repetition has shown the example of an examination of MONYOTOU -- this invention compound

in a test compound -- the -- the [I-1 table -] -- the compound number indicated to II-2 table showed.

The example 1 of an examination

The effect over a mandarin orange red spider

A mandarin orange HADANI (*Panonychus citri*) female imago is inoculated three animals for every piece of a leaf on the filter paper of the cup upper part which enabled it to compensate moisture with the piece of citrus leaves which does not go away the diameter of 1cm from a bottom, and it holds in the high temperature room of 25DEGC.

24 hours after, under a microscope, the number of laying eggs is investigated and united and additional inoculation of the two healthy insects is carried out for every piece of a leaf.

The water diluted solution (250 ppm) of the water-dispersible agent of the test compound adjusted according to the example 2 of a tablet is sprinkled by the glass spray, and it holds to 25DEGC.

Two days after medical fluid processing, life and death were judged under the microscope and it asked for the rate of insect killing (%).

The number of test insect was made into 20 female imagos.

Moreover, it asked for egg-killing rate (%) to the egg carried out under birth in after [processing] two days before medical fluid processing after processing on the 10th.

In addition, the number of test egg was used as 40 or more eggs at the time of processing.

the result -- the -- it was shown in the III table.

the -- an III table

| 化合物 番 号 | 殺卵率% — 殺虫率% | 化合物 番 号 | 殺卵率% — 殺虫率% | 化合物 番 号 | 殺卵率% — 殺虫率% |
|------------|----------------|------------|----------------|------------|----------------|
| (1) | 100/100 | (23) | 100/100 | (45) | 100/ 71 |
| (2) | 100/100 | (24) | 100/100 | (46) | 100/100 |
| (3) | 100/100 | (25) | 100/100 | (47) | 100/ 43 |
| (4) | 100/100 | (26) | 100/100 | (48) | 100/ 86 |
| (5) | 100/100 | (27) | 100/100 | (49) | 100/ 0 |
| (6) | 100/ 78 | (28) | 100/100 | (50) | 98/ 33 |
| (7) | 100/ 67 | (29) | 100/100 | (51) | 100/100 |
| (8) | 100/ 78 | (30) | 100/ 0 | (52) | 100/ 78 |
| (9) | 100/100 | (31) | 100/100 | (53) | 100/100 |
| (10) | 100/100 | (32) | 100/ 38 | (54) | 100/100 |
| (11) | 100/100 | (33) | 100/ 78 | (55) | 100/ 0 |
| (12) | 100/100 | (34) | 100/100 | (56) | 100/100 |
| (13) | 77/ 0 | (35) | 100/100 | (57) | 100/ 86 |
| (14) | 100/100 | (36) | 100/100 | (58) | 100/100 |
| (15) | 100/100 | (37) | 100/ 11 | (59) | 100/100 |
| (16) | 93/ 0 | (38) | 100/ 67 | (60) | 100/ 86 |
| (17) | 100/ 78 | (39) | 100/ 0 | (61) | 100/100 |
| (18) | 100/100 | (40) | 100/ 0 | (62) | 100/100 |
| (19) | 100/100 | (41) | 100/ 0 | (63) | 100/100 |
| (20) | 100/100 | (42) | 100/ 0 | (64) | 100/100 |
| (21) | 100/100 | (43) | 91/ 0 | (65) | 100/100 |
| (22) | 100/100 | (44) | 100/ 0 | (66) | 100/100 |

| 化合物 番 号 | 殺卵率% — 殺虫率% | 化合物 番 号 | 殺卵率% — 殺虫率% | 化合物 番 号 | 殺卵率% — 殺虫率% |
|------------|----------------|------------|----------------|------------|----------------|
| (67) | 100/ 25 | (91) | 98/ 25 | (116) | 85/ 0 |
| (68) | 99/100 | (92) | 100/100 | (117) | 100/ 90 |
| (69) | 100/ 88 | (93) | 100/100 | (118) | 100/ 90 |
| (70) | 100/100 | (94) | 100/100 | (119) | 100/ 90 |
| (71) | 100/100 | (95) | 100/100 | (120) | 90/ 0 |
| (72) | 100/100 | (96) | 100/100 | (121) | 99/ 13 |
| (73) | 100/100 | (97) | 100/ 80 | (122) | 100/ 25 |
| (74) | 88/ 0 | (98) | 100/100 | (123) | 100/ 70 |
| (75) | 99/ 78 | (99) | 100/ 4 | (124) | 90/ 0 |
| (76) | 100/100 | (100) | 100/ 56 | (125) | 100/ 10 |
| (77) | 100/ 89 | (101) | 100/ 78 | (127) | 88/ 25 |
| (78) | 100/100 | (102) | 100/100 | (128) | 100/100 |
| (79) | 100/100 | (103) | 100/ 22 | (129) | 89/ 0 |
| (80) | 88/ 0 | (104) | 100/ 60 | (130) | 100/100 |
| (81) | 100/ 88 | (105) | 100/ 40 | (131) | 88/ 0 |
| (82) | 100/100 | (106) | 100/100 | (132) | 100/ 78 |
| (83) | 100/ 88 | (107) | 100/ 89 | (133) | 100/100 |
| (84) | 100/100 | (108) | 100/ 78 | (134) | 100/ 13 |
| (85) | 100/100 | (109) | 100/ 78 | (135) | 100/ 78 |
| (86) | 87/ 78 | (110) | 100/ 50 | (136) | 100/ 0 |
| (87) | 100/ 88 | (112) | 92/ 10 | (137) | 100/ 0 |
| (88) | 100/ 63 | (113) | 94/ 10 | 無処理 | 0/ 0 |
| (89) | 100/100 | (114) | 100/ 10 | | |
| (90) | 100/100 | (115) | 100/100 | | |

The example 2 of an examination

The effect over HIMETOBIUNKA

After having diluted the emulsion of the test compound adjusted according to the example 1 of a tablet to 50 ppm, immersing rice seedling before and behind 5 leaf terms in this diluted solution

and making a medical fluid absorb, it blow dry(ed) and held in the test tube which put little water into the bottom.

It inoculated the HIMETOBIUNKA (*Laodelphax striatellus*) imago ten animals at a time in the test tube, and held by 25DEGC.

It asked for the rate of insect killing (%) 48 hours after processing.

the result -- the -- it was shown in IV table.

the -- IV table

| 化合物 番 号 | 殺虫率 (%) | 化合物 番 号 | 殺虫率 (%) |
|------------|------------|------------|------------|
| (1) | 100 | (24) | 80 |
| (4) | 80 | (25) | 90 |
| (6) | 80 | (29) | 100 |
| (7) | 100 | (31) | 95 |
| (8) | 100 | (45) | 95 |
| (9) | 100 | (47) | 95 |
| (10) | 100 | (48) | 100 |
| (12) | 90 | (50) | 100 |
| (13) | 90 | (51) | 100 |
| (19) | 85 | (65) | 100 |
| (20) | 100 | (67) | 85 |
| (21) | 85 | (68) | 100 |
| (22) | 80 | (72) | 100 |

| 化合物 番 号 | 殺 虫 率 (%) | 化合物 番 号 | 殺 虫 率 (%) |
|------------|--------------|------------|--------------|
| (73) | 100 | (87) | 100 |
| (75) | 75 | (88) | 100 |
| (76) | 95 | (89) | 100 |
| (77) | 85 | (90) | 100 |
| (78) | 80 | (92) | 90 |
| (79) | 95 | (93) | 80 |
| (80) | 85 | (94) | 85 |
| (81) | 100 | (97) | 95 |
| (82) | 100 | (103) | 85 |
| (83) | 95 | (109) | 95 |
| (84) | 95 | (115) | 90 |
| (85) | 100 | (126) | 80 |
| (86) | 80 | 無处理 | 0 |

The example 3 of an examination

a lotus -- the effect over MONYOTOU

The bottom and the upper part of a laboratory dish with a diameter of 9cm are covered with filter paper, respectively, and 2ml of 100 ppm diluted solutions of a test compound water-dispersible agent shown in the example 2 of a tablet is uniformly dropped at this with a pipette.

On upside filter paper, artificial bait is attached beforehand.

if it is neglected for about 1 hour and filter paper dries moderately -- a bottom -- a lotus -- it inoculated ten MONYOTOU (*Spodoptera litura*) 1 age larva at a time, and held to 25DEGC.

The rate of insect killing (%) was investigated 48 hours after processing.

The result was shown in the Vth table.

The Vth table

| 化合物 番 号 | 殺虫率 (%) | 化合物 番 号 | 殺虫率 (%) |
|------------|------------|------------|------------|
| (1) | 100 | (64) | 100 |
| (2) | 80 | (65) | 80 |
| (3) | 100 | (68) | 100 |
| (7) | 100 | (69) | 100 |
| (8) | 100 | (72) | 95 |
| (9) | 100 | (73) | 90 |
| (10) | 100 | (75) | 100 |
| (12) | 93 | (76) | 95 |
| (20) | 85 | (77) | 100 |
| (21) | 100 | (78) | 80 |
| (22) | 95 | (81) | 100 |
| (29) | 100 | (82) | 100 |
| (31) | 100 | (83) | 80 |
| (45) | 85 | (109) | 95 |
| (48) | 95 | (130) | 95 |
| (51) | 100 | 無処理 | 0 |

The example 4 of an examination

The effect over KONAGA

The Japanese radish which sprout(ed) in the ice cream cup beforehand was prepared, and 3 age larva of KONAGA (*Plutella xylostella*) were inoculated into this.

2ml (250 ppm) of water diluted solutions of the emulsion of the test compound prepared according to the example 1 of a tablet using the spraying pipe was sprinkled by the glass spray.

It investigated after medical fluid processing on the 3rd, and asked for the rate of insect killing (%).

In addition, the number of test insect was made into ten per the 1st division.

the result -- the -- it was shown in VI table.

the -- VI table

| 化合物 番 号 | 殺虫率 (%) | 化合物 番 号 | 殺虫率 (%) |
|------------|------------|------------|------------|
| (1) | 100 | (29) | 100 |
| (2) | 100 | (30) | 98 |
| (4) | 97 | (31) | 100 |
| (5) | 100 | (34) | 100 |
| (6) | 80 | (35) | 91 |
| (7) | 100 | (36) | 91 |
| (9) | 100 | (37) | 89 |
| (10) | 98 | (41) | 97 |
| (14) | 100 | (45) | 98 |
| (15) | 92 | (46) | 100 |
| (16) | 98 | (51) | 99 |
| (18) | 100 | (53) | 92 |
| (19) | 96 | (54) | 97 |
| (20) | 100 | (56) | 95 |
| (21) | 97 | (58) | 96 |
| (22) | 100 | (59) | 84 |
| (23) | 100 | (64) | 100 |
| (24) | 97 | (65) | 97 |
| (26) | 81 | (66) | 100 |
| (27) | 97 | (68) | 76 |

| 化合物 番 号 | 殺虫率 (%) | 化合物 番 号 | 殺虫率 (%) |
|------------|------------|------------|------------|
| (72) | 89 | (103) | 89 |
| (73) | 95 | (106) | 100 |
| (76) | 92 | (107) | 100 |
| (78) | 94 | (108) | 100 |
| (88) | 97 | (109) | 100 |
| (89) | 96 | (110) | 92 |
| (90) | 95 | (114) | 85 |
| (93) | 96 | (115) | 96 |
| (96) | 100 | (128) | 97 |
| (99) | 95 | (129) | 87 |
| (101) | 94 | (130) | 100 |
| (102) | 88 | 無处理 | 0 |

The example 5 of an examination

The activity comparison examination to various noxious insects

the test compound water-dispersible agent which carried out copper according to the example 2 of a tablet -- HIMETOBIUNKA (50 ppm) and a lotus -- it processed to MONYOTOU (100 ppm),

KONAGA (250 or 125 ppm), and mandarin orange HADANI (250 ppm), and the rate of insect killing and egg-killing rate were investigated.

the result -- the -- it was shown in the VII table.

the -- a VII table

| | ミカンハダニ | ヒメトビウンカ | | アゼモシロ | | コナガ | |
|--------------------|---------|-----------------|--------------|-----------------|------------|-----------------|-----------------|
| | | 施用濃度 50 ppm | 殺虫率 (%) | 施用濃度 100 ppm | 殺虫率 (%) | 施用濃度 250 ppm | 施用濃度 125 ppm |
| | | 施用濃度 250 ppm | 殺卵率% 殺虫率% | | | | |
| 本発明になる化合物(10) | 100/100 | | 100 | 100 | 98 | 85 | |
| | | | | | | | |
| 比較例 (例示化合物No.2) | 0/0 | | 0 | 0 | 35 | 10 | |
| | | | | | | | |

(Effect of invention)

This invention compound has an effect and the especially high egg-killing effect excellent in the lepidopteran noxious insect which damages vegetables, a fruit tree, a rice, etc., a half-wing noxious insect, and HADANI.

Representative
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